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Evaluation of the Performance of Various Amine Based Solvents in an Optimized Multipurpose Technology Development Pilot Plant

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Abstract The overall goal of this work was to show experimentally using a CO_2 capture plant of reasonable capacity the contributions of plant and solvent optimization to minimizing the steam requirement. Experimental studies were conducted in an optimized CO_2 capture pilot plant to evaluate the performance of RS-1 solvent and 5 molar aqueous MEA. The first objective was to test a highly improved plant configuration that can minimize both the amine circulation rate and the steam requirements below the values reported in the literature for a desired CO_2 recovery. The second objective was to show the superior performance of RS-1 relative to 5 molar MEA. Both these strategies were aimed at reducing the steam requirement without increasing the size or complexity of the CO_2 capture plant. The pilot plant used for the tests was the highly optimized multi-purpose technology development plant at the International Test Center for CO_2 capture which processes 186 Nm^3/h of flue gas to produce CO_2 with a nominal capacity of 1 tonne per day. This plant has 12-inch ID for the absorber and stripper columns. The inlet flue gas temperature was 40°C while the CO_2 concentrations in the flue gas were 4 and 8% based on exhaust gases obtained respectively from a natural gas turbine-natural gas boiler combination, and a natural gas boiler. The results show that for flue gas containing 4 and 8% CO_2 for a 90% recovery or absorber efficiency, the steam duties using our optimized plant configuration were respectively 2.03 and 1.43 kg steam/kg CO_2 for MEA as compared to industry and literature values based on conventional plant configuration in the range of 1.9 to 2.5 kg steam/kg CO_2 depending on the CO_2 concentration in the flue gas. When RS-1 solvent was used, the steam duties using the optimized plant configuration were respectively 1.74 and 1.35 kg steam/kg CO_2 which showed improvements over the corresponding performance of 5 molar MEA. It was interesting to observe that with a completely modified and optimized process configuration, the heat duty using RS-1 was 1kg steam/kg CO_2. These results show that in order to minimize steam requirements in a post combustion CO_2 capture amine plant, there is the need to optimize both the plant process configuration and the solvent.

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Keywords: *process configuration; solvent optimization; process optimization; steam requirements.*

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1. Introduction

Post combustion capture of CO_2 from combustion flue gases is typically performed using amine solvents. In the amine process, it is desired that the absorption is fast while the stripping requires as little energy as possible in order to minimize the operating costs. It has been indicated that the cost of steam used for stripping makes up about 50-70% of the operating cost. Also, it is well known that the steam requirement depends on both the plant design/configuration as well as the type of solvent used. The overall goal of this work was to show experimentally using a CO_2 capture plant of reasonable capacity the contributions of plant and solvent optimization to minimizing the steam requirement. Experimental studies were conducted in an optimized CO_2 capture pilot plant to evaluate the performance of RS-1 solvent and 5 molar aqueous MEA. The first objective was to test a highly improved plant configuration that can minimize both the amine circulation rate and the steam requirements below the values reported in the literature for a desired CO_2 recovery. The second objective was to show the superior performance of RS-1 relative to 5 molar MEA. Both these strategies were aimed at reducing the steam requirement without increasing the size or complexity of the CO_2 capture plant.

2. Experimental Section

Pilot Plant: The process description is typical of an amine solution extraction plant with specific features to accommodate research activities. Figure 1 represents a flow diagram of the facility before the upgrades. The boiler is a 250 KW high pressure steam boiler that provides high quality steam, and flue gas to the process skid. Also on the same skid is a 30 KW micro gas turbine. The configuration is designed such that the boiler can operate on its own or in conjunction with the micro turbine. When operating the micro turbine, a low CO_2 concentration feed is produced; therefore, the boiler is operated as a duct burner to enrich the CO_2 concentration of the flue gas while consuming the high excess O_2 content from the turbines exhaust. Additionally, the boiler provides the process steam required by the process.

The flue gas is carried by a 168.3 mm (6 in) pipe and is drawn into the inlet gas scrubber from a blower located downstream of the scrubber. This multi-purpose inlet gas scrubber is comprised of a cylindrical conical bottom tank complete with cooling coils and a 2.5 m (8.2 ft) packed section on top. Cooling water to the cooling coils is provided by the main physical plant chillers. The cooled water is circulated to the top of the packing both cleaning and cooling the gas before it exits the unit. From here the inlet feed gas blower boosts the gas pressure where it is then measured before entering the absorber column. As previously mentioned, there are three absorber columns each composing of three (3) 323.8 mm (12 in) diameter sections for a total height of 10 m (32.8 ft). Any of the columns can be selected through the use of isolation valves located at the inlet, outlet, and amine inlet to the vessel. Additionally, the columns are designed such that the amine can enter into a column at any one of the three sections as illustrated in Figure 1. The columns are also equipped with temperature measurement every 0.6 m (2 ft), sample gas measurement at the same interval, and provisions for corrosion coupon measurement. Through the use of the data acquisition system, a temperature profile can be viewed on the computer screen. Additionally, a sample gas profile is achieved for display via a switching valve controlled by the computer control system and analyzed by a single CO_2/O_2 analyzer. There are also CO_2/O_2 analyzers on the gas inlet as well as the gas outlet to the absorber. Amine pumped to the absorber column from the lean amine storage tank is in counter flow with the feed gas. As it descends down through the packing it picks up CO_2 and accumulates in the rich amine surge vessel. The off-gas enters the off-gas scrubber before it exits back to the atmosphere. The construction of the off-gas scrubber is typical of the inlet gas scrubber except along with cooling the gas, the purpose is to recover any amine that might be entrained in the amine and return it back to the process. The rich amine is pumped through the lean/rich heat exchanger where it picks up heat before entering near the top of the stripper column. This 323.8 mm (12 in) diameter by 10 m (32.8 ft) tall column comprises of two sections each filled with the Flexipac 700Y structured packing material along with a 0.6 m (2 ft) packed reflux section at the very top. A reboiler located near the bottom and interconnected with the stripper provides the heat medium necessary to strip out the CO_2 from the amine. The design of the reboiler is to operate at 90 – 97 kPag (13 – 14 psig) and 121 °C (250 °F). Stripping steam is produced which passes upward through the stripper column. As the rich amine descends in a counter flow, CO_2 is stripped out of the amine which reverses the amine absorption reaction. The stripped lean amine accumulates in the reboiler where it exits via the lean amine return pump through the lean/rich exchanger followed by the lean amine cooler and ultimately back to the amine storage tank. From the top of the stripper column, CO_2 product and water vapour enter the reflux condenser where the majority of the water is condensed, accumulated in the reflux accumulator and pumped back to the top of the stripper column reflux section. The CO_2 product is measured and passes through a back pressure valve which is used to maintain stripper and reboiler operating pressure. The CO_2 product then enters a CO_2 wash scrubber which is typical of both the inlet gas and off-gas scrubbers except smaller in size. Additionally, an independent chiller is used to provide the cooling medium in order to cool down the CO_2 gas to a desired temperature of 4 ^oC (39.2 ^oF). From here the gas can either exit back to the atmosphere or pass through a dryer and purification unit in order to produce food grade quality CO_2. This unit consists of a dryer unit containing silica gel and an activated carbon unit.

An additional item on the process skid is the reclaimer that is used to regenerate degraded amine due to an excess of heat stable salts (HSS) and other impurities in the amine solution. Heat and caustic solution injection are the prime process ingredients for the regeneration of the amine. With a slip stream of 5% lean amine into the reclaimer as illustrated in the process flow diagram, the introduction of heat and caustic releases the amine from the degradation product as a vapour back into the stripper leaving behind a non-regenerable sludge at the bottom of the reclaimer which can be collected and disposed of.

The heat medium for both the reboiler and reclaimer is the steam from the boiler system. Due to the amount of feed gas required by the process skid, the boiler produces an excess amount of steam. In order to handle this excess steam, waste heat coils are utilized. Flow measurement is provided to the reboiler, reclaimer, and waste heat coils. The pressure of the boiler is maintained by controlling the amount of heat wasted in the waste heat coils. This equipment is also equipped with traps to ensure the pressure of steam is controlled. Condensate from the traps enters a flash tank where low pressure steam is formed for use in certain dryers within the process skid.

Analysis of the samples (fresh and used) was carried out using a gas chromatography-mass spectrometer (GC/MS model HP 6890/5073 supplied by Hewlett-Packard Canada Ltd., Montreal Quebec, Canada). An HP – Innowax column (length = 30m, internal diameter = 250μm, thickness = 0.25μm) packed with cross - linked – poly – ethylene glycol was used in the GC for the separation of components. These components were identified by their mass spectra. Prior to GC/MS analysis, each sample was diluted with deionized water to five times its original volume to avoid column overload and to improve separation of the components. Sample injection into the GC column was done using an auto – injector (model 7683) supplied by Hewlett – Packard Ltd. The exact concentration of fresh aqueous MEA and MDEA was obtained by titrating known volumes of MEA/MDEA with 1N HCl using 0.1wt% methyl orange solution as indicator. Several trials were done to select the optimum operating conditions for the GC/MS. The conditions used are summarized as follows. An autoinjector (model 7683, supplied by Hewlett-Packard) was used to automatically introduce samples into the GC column to give better reproducibility. A 10μL syringe with an injection volume of 0.2μL was used and a split mode was selected for the inlet with a split ratio of 10:1, split flow of 10.3 mL/min and total flow of 13.9 mL/min. The inlet temperature and pressure were 70^oC and 9.18 psi, respectively. The initial temperature of the oven was 100^oC with a hold time of 0mins while the final temperature was 240^oC with a hold time of 10 min with an oven ramp of 10^oC/min for a total run time of 27 min. The column flow rate was 1mL/min while the pressure and average velocity were 9.18 psi and 37 cm/sec. For the MS parameters, the interface, quadruple and source temperatures were 250, 150 and 230^oC, respectively and the electron multiplier (EM) voltage was 1200V. The error of the GC/MS was estimated to be less than ±3%. Majority of the samples were analyzed two or more times to check the reproducibility of analysis. The error was also less than ±2%. MEA and MDEA concentrations were based on peak area% only, and thus, represented relative concentrations. The absolute molar concentrations were obtained with HPLC with a refractive index detector (Agilent Technologies Canada, Mississauga, Ontario, Canada) using a nucleosil column with a phosphate buffer. The CO_2 loading in the lean and rich amine samples were determined using a Chittick CO_2 analyzer.

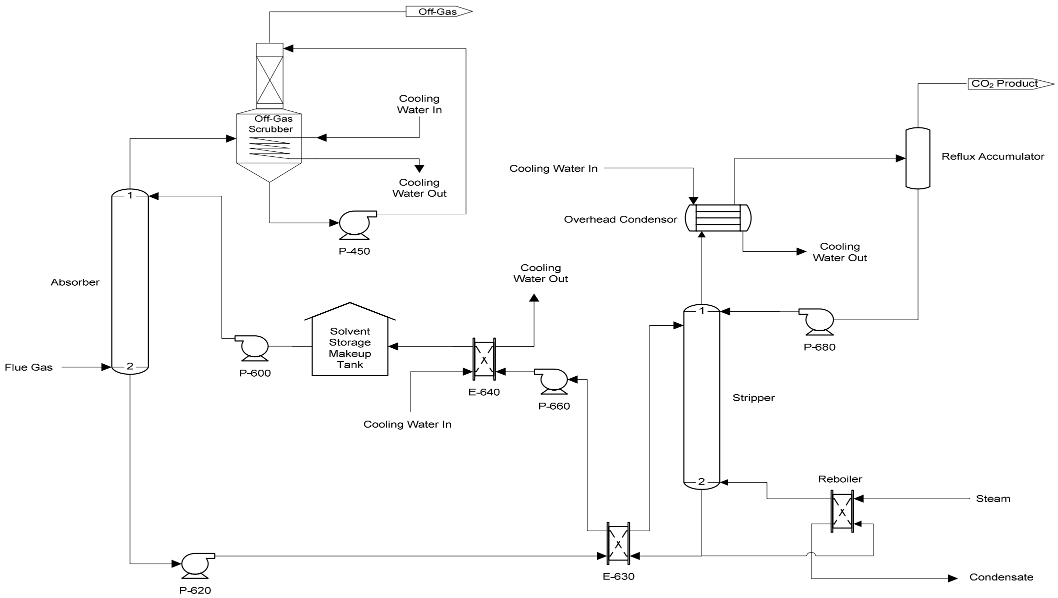

Figure 1: Process Flow Diagram of the Optimized UR Multipurpose CO_2 capture Plant

The heat duty for regeneration was evaluated by making an energy balance around the reboiler and/or the stripping column. In the BD column, the heat used in the reboiler for stripping was supplied by a glycol heater whereas that for the UR plant was supplied by a steam boiler.

Specific Conditions

A number of optimized tests were performed to determine the optimum operating parameters for 5 molar MEA and RS-1 based on the utilization of a combined micro turbine and boiler exhaust to supply a flue gas stream with CO_2 concentrations of 4% and 8% with high oxygen concentration. Additionally, operating conditions were varied utilizing 3 separate steam rates to the reboiler and 3 separate inlet gas temperatures to the absorber. In the later set of tests, all other conditions such as amine circulation rate, reboiler pressure, and feed gas supply rate were fixed.

The absorber column is comprised of 3 sections packed with structured packing. Each section is approximately 2.9 meters which includes the packing support, packing material, and distribution. The total packing height in each section is approximately 2.19 meters. Also in each section there are 5 sample points for obtaining temperatures, sample gas concentrations, and liquid collection for a total of 15 sample points across the column. For the 4% CO_2 inlet case, the solvent was injected in the bottom two sections of the column (for 5 molar MEA and RS-1 only). For the 8% CO_2 case for all the two solvents, all three sections of the column were utilized.

3. **Results and Discussion**

The results for the three categories of test (i.e. for 5 molar MEA, RS-1 and optimized modified process configurations are presented in Tables 1-3. The results show that for flue gas containing 4 and 8% CO_2 for a 90% recovery or absorber efficiency, the steam duties using our optimized plant configuration were respectively 2.03 and 1.43 kg steam/kg CO_2 for MEA as compared to industry and literature values based on conventional plant configuration in the range of 1.9 to 2.5 kg steam/kg CO_2 depending on the CO_2 concentration in the flue gas. When RS-1 solvent was used, the steam duties using the optimized plant configuration were respectively 1.74 and 1.35 kg steam/kg CO_2 which showed improvements over the corresponding performance of 5 molar MEA. It was interesting to observe that with a completely modified and optimized process configuration, the heat duty using RS-1 was 1kg steam/kg CO_2. These results show that in order to minimize steam requirements in a post combustion CO_2 capture amine plant, there is the need to optimize both the plant configuration and the solvent.

Table 1: Solvent Optimization – Case Scenario for 5 molar MEA

Inlet CO_2 Concentration	8	4	%
Absorber Efficiency	90.15	90.67	%
Amine Circulation Rate	7.0	3.9	kg/min
CO_2 Production	0.56	0.28	tonne/day
Steam Rate	33	24	kg/hr
Heat Duty	56,845	81,147	Btu/lb-mole CO_2
Steam Input	1.65	2.03	kg steam/kg CO_2
Lean Loading	0.228	0.179	mol/mol
Rich Loading	0.429	0.409	mol/mol

Table 2: Solvent Optimization – Case Scenario for RS_1

Inlet CO_2 Concentration	8	4	%
Absorber Efficiency	90.60	90.50	%
Amine Circulation Rate	7.0	3.50	kg/min
CO_2 Production	0.56	0.29	tonne/day
Steam Rate	33	21.02	kg/hr
Heat Duty	53,952	69,250	Btu/lb-mole CO_2
Steam Input	1.35	1.74	kg steam/kg CO_2
Lean Loading	0.228	0.188	mol/mol
Rich Loading	0.429	0.404	mol/mol

		Heat Duty (BTU/lb-mole)	Lean Loading (mol/mol)	Rich Loading (mol/mol)	CO_2 production (tonne/day)	Steam Input (kg steam/kg CO_2)
5 molar MEA	Simulation	55,590	0.2504	0.4837	0.57	1.30
	Experiment	48,924 ± 5436	0.2270	0.5024	0.58	1.21 ± 0.14
RS-1	Simulation	43,733	0.2321	0.4222	0.53	1.04
	Experiment	39231 ± 5117	0.1835	0.4252	0.58	0.98 ± 0.17

4. Conclusions

Experimental studies were conducted in an optimized CO_2 capture pilot plant to evaluate the performance of RS-1 solvent and 5 molar aqueous MEA. The first objective was to test a highly improved plant configuration that can minimize both the amine circulation rate and the steam requirements below the values reported in the literature for a desired CO_2 recovery. The second objective was to show the superior performance of RS-1 relative to 5 molar MEA. Both these strategies were aimed at reducing the steam requirement without increasing the size or complexity of the CO_2 capture plant. The pilot plant used for the tests was the highly optimized multi-purpose technology development plant at the International Test Center for CO_2 capture which processes 186 Nm^3/h of flue gas to produce CO_2 with a nominal capacity of 1 tonne per day. This plant has 12-inch ID for the absorber and stripper columns. The inlet flue gas temperature was 40°C while the CO_2 concentrations in the flue gas were 4 and 8% based on exhaust gases obtained respectively from a natural gas turbine-natural gas boiler combination, and a natural gas boiler. The results show that for flue gas containing 4 and 8% CO_2 for a 90% recovery or absorber efficiency, the steam duties using our optimized plant configuration were respectively 2.03 and 1.43 kg steam/kg CO_2 for MEA as compared to industry and literature values based on conventional plant configuration in the range of 1.9 to 2.5 kg steam/kg CO_2 depending on the CO_2 concentration in the flue gas. When RS-1 solvent was used, the steam duties using the optimized plant configuration were respectively 1.74 and 1.35 kg steam/kg CO_2 which showed improvements over the corresponding performance of 5 molar MEA. It was interesting to observe that with a completely modified and optimized process configuration, the heat duty using RS-1 was 1kg steam/kg CO_2. These results show that in order to minimize steam requirements in a post combustion CO_2 capture amine plant, there is the need to optimize both the plant process configuration and the solvent.